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Mechanical Behavior of Tantalum and Tantalum-Tungsten Alloys: Texture Gradients and Macro/Micro-Response

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Abstract

We have examined the mechanical response of unalloyed tantalum and tantalum-tungsten alloy annealed plates over a wide range of loading conditions. In general, we have observed that unalloyed tantalum exhibits non-uniform mechanical behavior, for example, hourglassing of compression samples and multiple instabilities during tensile deformation. In contrast, the tantalum-tungsten alloys do not exhibit any unusual non-uniform behavior. In this work, we present data revealing the spatial distribution of texture in unalloyed tantalum and tantalum-tungsten alloys. Significant variations in texture both through the thickness and from one area of the plate to another were found to be characteristic of the unalloyed tantalum. The dominant feature of the texture variations was found to be enhanced <111> crystal direction fractions at the center of the plate, with a decreasing fraction near the surface. We find that the variation in texture in the tantalum-tungsten alloys is substantially less than that seen in the unalloyed tantalum with primarily a <100> cube texture throughout. This study suggests that the texture gradients are responsible for the non-uniform mechanical response of unalloyed tantalum and that the uniform behavior of the tantalum-tungsten alloys is a consequence of the absence of texture gradients.

1. Introduction

Many studies have addressed the macroscopic mechanical response of unalloyed tantalum [1-3] and tantalum-tungsten alloys [4,5] over a wide range of deformation conditions. In general, pure tantalum exhibits mechanical behavior typical of bcc metals with flow stress being extremely sensitive to strain-rate and temperature. The addition of tungsten as an alloying element causes significant changes in mechanical behavior with an increase in yield stress, increase in work hardening, and a decrease in strain rate and temperature sensitivity. Researchers have observed that the mechanical behavior of tantalum-tungsten alloys resembles that of fcc metals [4].

One of the outstanding issues associated with the mechanical response of ingot metallurgy (IM) rolled and annealed tantalum plate is the non-uniform behavior observed in a variety of deformation modes. For example, it is typical to observe "hourglassing" or inverse barreling in the cross sections of deformed compression samples. Also, multiple "necks" are sometimes observed in tensile samples. These types of non-uniformities in mechanical behavior are also observed in unalloyed vanadium, also a bcc metal. In contrast, none of these unusual behaviors are observed in the tantalum-tungsten alloys.

In a study reported by Wright et al.[6], the spatial distribution of texture in unalloyed tantalum was characterized using Orientation Imaging Microscopy (OIM). This scanning electron microscopy technique allows determination of grain size, microtexture, and grain boundary character distribution. Significant variations of the texture were documented showing predominantly <100> cube texture found at, and near the surfaces with a predominantly <111> texture found in the center region. In subsequent work, Wright et al. performed a numerical simulation of compression deformation of unalloyed tantalum plate considering the measured variation in texture [7]. The constitutive relationship used followed the Taylor crystal plasticity theory. Good qualitative agreement between experiment and the simulation was reported in terms of capturing the hourglass shape, suggesting that these shapes are due to the observed texture gradients.

Lawrence Livermore National Laboratory (LLNL) has examined the uniaxial stress-strain compression response of IM, rolled and annealed, tantalum and tantalum-tungsten plate stock of many different lots of material under the now defunct Balanced Technology Initiative (BTI) program. In this program it was observed that the unalloyed tantalum exhibited, without exception, non-uniform mechanical behavior. In this paper, we present some examples of mechanical behavior which illustrate the non-uniform response of unalloyed tantalum and, in stark contrast, uniform mechanical response of the tantalum-tungsten alloys (nominally 2.5, 5, and 10 wt.% tungsten). The spatial distribution of texture in unalloyed tantalum and the tantalum-tungsten alloys was characterized using OIM. We also discuss data revealing significant variations in texture in unalloyed tantalum, and relatively uniform texture in the tungsten containing alloys.

2. Mechanical Response

2.1 Compression

Unalloyed tantalum and tantalum-tungsten plate stock with 2.5, 5, and 10 wt.% tungsten, nominally 6 mm in thickness was provided for this study by Cabot Corp., Boyertown, PA. Details of the IM processing are described by Michaluk [8]. The compression stress-strain response of the study materials was found to be, in general, consistent within a given plate. For example, the variation in flow stress at a given value of compression strain is generally within 5% of the flow stress. However, unalloyed tantalum exhibits some unusual non-uniform mechanical behavior on a length scale equal to a fraction of the plate thickness. For example, Fig. 1 shows inverse barrel or hourglass shapes, a phenomenon observed without exception in all the compression experiments performed on unalloyed tantalum over a wide range of strain rates (10^{-5} - 10^{4} s^{-1}) and test temperatures

(77 - 400K). We have also observed a significant variation in the degree of non-uniformity; in some samples, the hourglass shape is uniform and in others the hourglass shape tends to be localized toward one end of the sample. This inhomogeneous mechanical response is not observed to any extent in the tantalum-tungsten alloys that have been studied at LLNL. In contrast with unalloyed tantalum, the tantalum-tungsten alloys exhibit deformation behavior which is typical; some slight, but uniform, barreling of the compression sample is observed at a compression strain of 30%, as shown in Fig. 1.

The hourglassing effect during compression led to an investigation of the mechanical response of the plate material as a function of position through the thickness. Small samples were conventionally machined from two regions in a plate: near the surface and spanning the centerline. The stress-strain response of samples indicated the yield points of the samples from both regions were essentially equal. However, the work hardening in the center region appeared to be significantly higher, leading to an increase in flow stress of about 15% relative to the surface region at a strain of 0.30, as shown in Fig. 2. This appears to be consistent with the noted hourglassing phenomenon, i.e., the center region has higher strength. A study of microhardness was also conducted, but failed to reveal any statistical variation in hardness. This may be due to the integral nature of the hardness test in that yield strength and work hardening behavior both have effects on the measured hardness number.

The variation of impurity concentrations in the plate at different locations was also investigated because of the pronounced effect of interstitials on mechanical response of bcc metals. Samples analyzed from the center and surface areas of the plate were found to be essentially identical with the exception of oxygen and carbon, which were found to be higher in the surface region. Table 1 summarizes these results. Clearly, the hourglassing effect cannot be related to this variation because of the hardening effects of interstitial impurities in bcc materials.

Table 1. Chemical analysis of tantalum plate near the surface and the centerline.

wt ppm	O	N	C	S	Nb	Mo	W
Ta surface	38	13	9	<1	170	15	60
Ta center	21	15	7	<1	175	16	60
2.5 W surface	46	10	21	<1	830	17	2.4%
2.5 W center	28	<2	7	<1	840	16	2.4%
5 W surface	42	14	4	<1	830	22	4.4 %
5 W center	63	21	7	<1	800	7.7	4.4 %
10 W surface	76	8	12	≤1	205	4.3	10.1 %
10 W center	27	4	7	<1	225	4.6	10.1 %

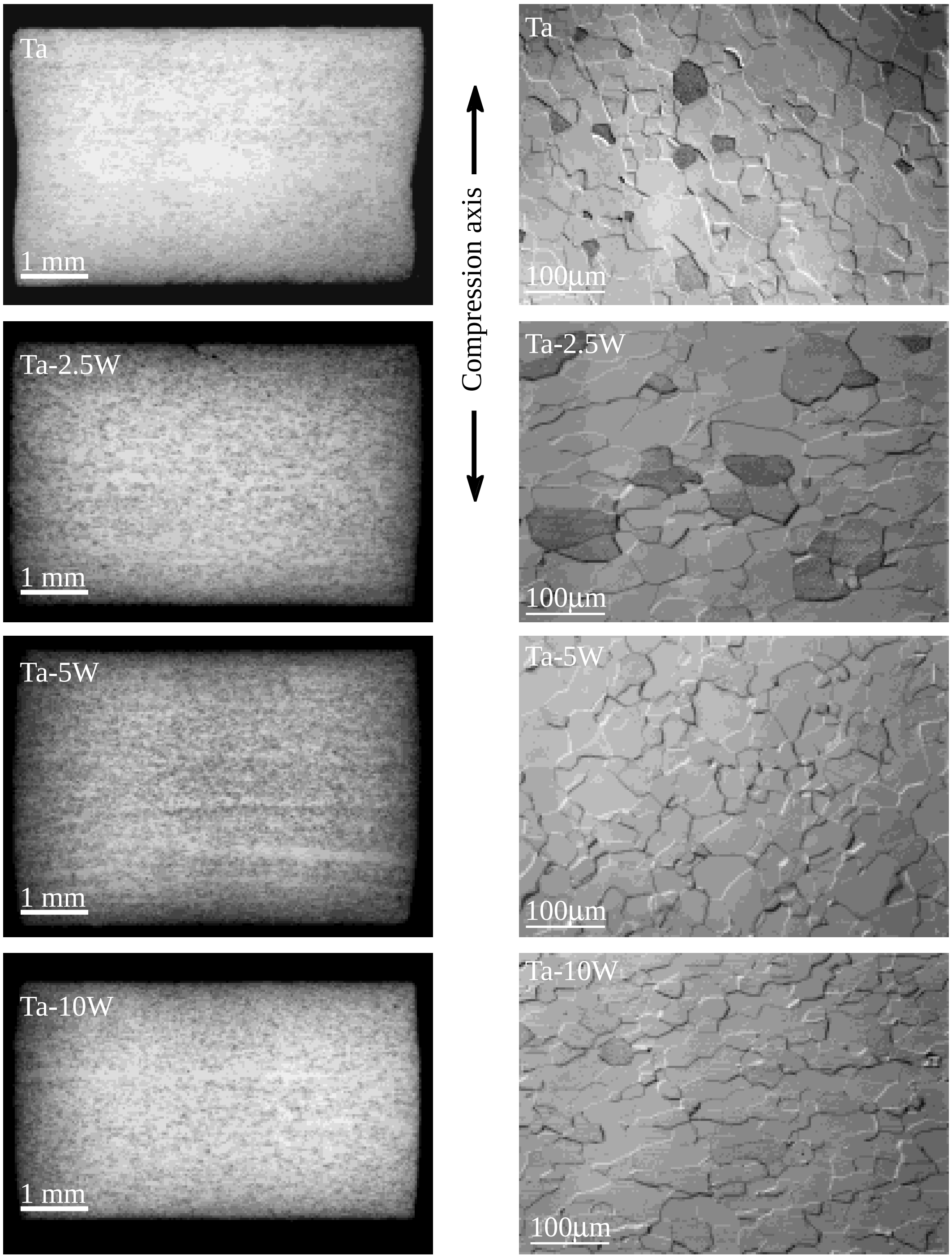


Figure 1. Microstructures and cross sections of compression samples deformed approximately 30% at a strain rate of 10^{-1} s^{-1}. The irregular hourglass shape was observed in all of the deformed Ta test materials; the uniform deformation (slight barreling) is common to all of the Ta-W alloys tested. Etchant: 100 ml deionized water, 20 ml powder ammonium bifluoride, and 50 ml nitric acid.

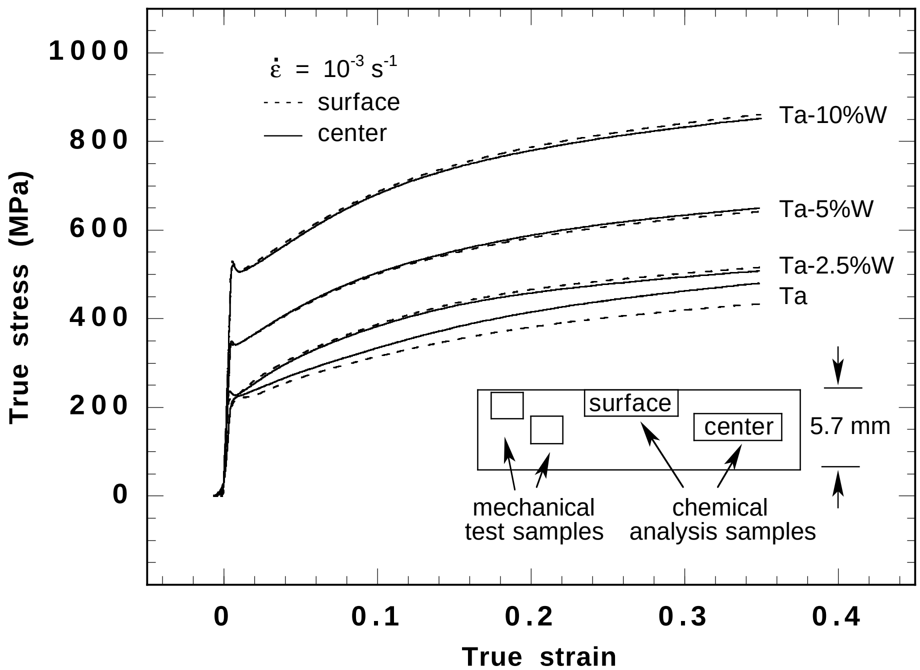


Figure 2. Compression response of unalloyed tantalum in center and surface regions.

2.2 Deformation Stability and Failure

Experiments at LLNL under the BTI program to examine the deformation stability of tantalum and tantalum-tungsten alloys included quasistatic and dynamic tensile tests. The behavior of tantalum under quasistatic tensile loading was found to be unusual in that approximately 30% of the samples tested exhibited two or more regions of instability (necks) prior to failure. Experiments performed at Alliant Tech Systems also revealed this behavior in unalloyed tantalum, but not in tantalum-tungsten alloys [9]. A sample which had two necks was sectioned and metallographically prepared to examine the microstructure. Significant variations in the grain structure, on the order 100 µm, appeared to be associated with the neck regions.

To examine the deformation stability and failure of materials under dynamic mixed mode loading, we performed a “punch-through” shear test based on the split Hopkinson pressure bar experiment. This experiment deforms an annular gage section in a combination of shear and extension. Figure 3 is a schematic diagram of the experiment and the sample geometry used to test the unalloyed Ta, Ta-2.5%W, and Ta-10%W. Figure 4 shows that the deformation and failure of the unalloyed tantalum was non-axisymmetric; in contrast, the Ta-W alloys exhibited uniform, axisymmetric deformation and failure. The results of these tests are consistent with the observation of double necks in tension testing and indicate that the irregular deformation behavior of unalloyed tantalum is eliminated by tungsten alloying additions.

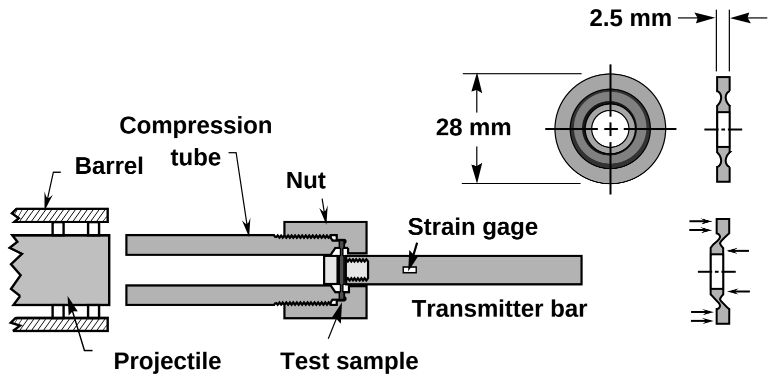


Figure 3. Schematic diagram of the dynamically loaded “punch-through” shear experiment and test sample. The experiment is a variation of the split Hopkinson pressure bar experiment. The strain rate in the gage section is on the order of 10^3 - 10^4 s^{-1}.

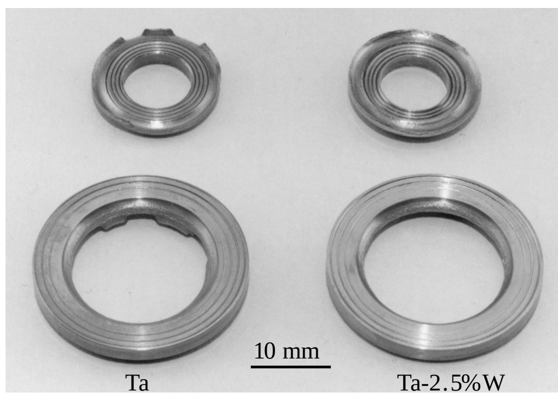


Figure 4. Photographs of failed Ta and Ta-2.5%W “punch-through” samples showing the irregular failure behavior of the unalloyed Ta sample.

3.0 Microtexture Analyses

The distribution of local orientation (microtexture) in unalloyed tantalum and tantalum-tungsten alloy plate samples was characterized using OIM [10]. This technique is based on automatic indexing of the electron backscatter Kikuchi diffraction patterns that are generated in a scanning electron microscope (SEM). Spatially specific crystallographic orientation measurements are made at discrete points in the microstructure, as prescribed by the user. Orientation images, or maps, are then generated by plotting the crystal orientation onto a color or gray scale, according to a user-defined aspect of the crystal orientation.

In this study, we generated maps to show the alignment of specific crystal directions with the plate normal. For example, a <111> crystal direction map is generated as follows: a point on the measurement grid is shaded gray if the lattice associated with the point is oriented so that the <111> crystal axis is aligned within 15° of the plate normal. The closer the alignment between the <111> crystal direction and the plate normal direction, the darker the shade of gray. These maps are useful for delineating texture gradients or inhomogeneous texture banding as well as identifying any relationships between the microtexture and grain-size banding. In this study, multiple scans of plate cross sections were made either near the surface or spanning the centerline of the plates. In addition to the crystal direction maps, pole figures were generated relative to the plate normal to characterize the overall texture.

3.1 Pure Ta

A total of 10 scans were performed on two unalloyed tantalum plate samples with a total of 368,701 data points covering an area of 38 mm^2 in 10 or 12 μm steps. Significant variations in microtexture were found to be typical within a given scan and also among the different scans along the plate. However, several general characteristics were observed: at the center regions, <111> direction fractions were found to dominate the microstructure with a decrease in <111> near the surface as shown in Fig. 5(a). The <100> crystal direction fractions were found to be very low near the center and slightly enhanced near the surface, as shown in Fig. (b). This result is in contrast to those of previous investigations of microtexture in IM unalloyed tantalum [7] which showed a high fraction of <100> normals near the surface. The pole figures calculated from all of the OIM scans indicated a relatively strong <111> fiber texture along with a weak <100> cube texture.

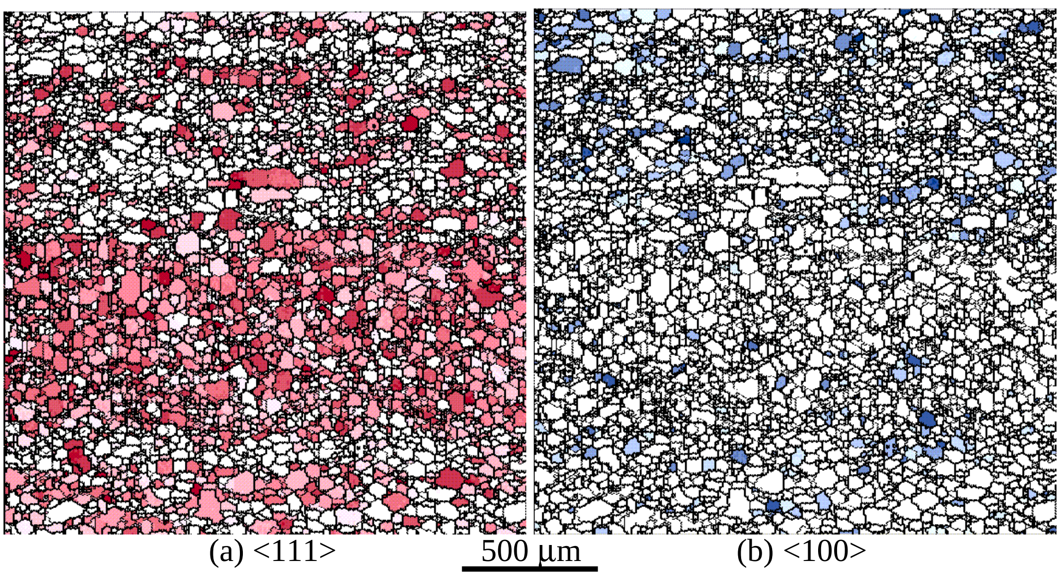
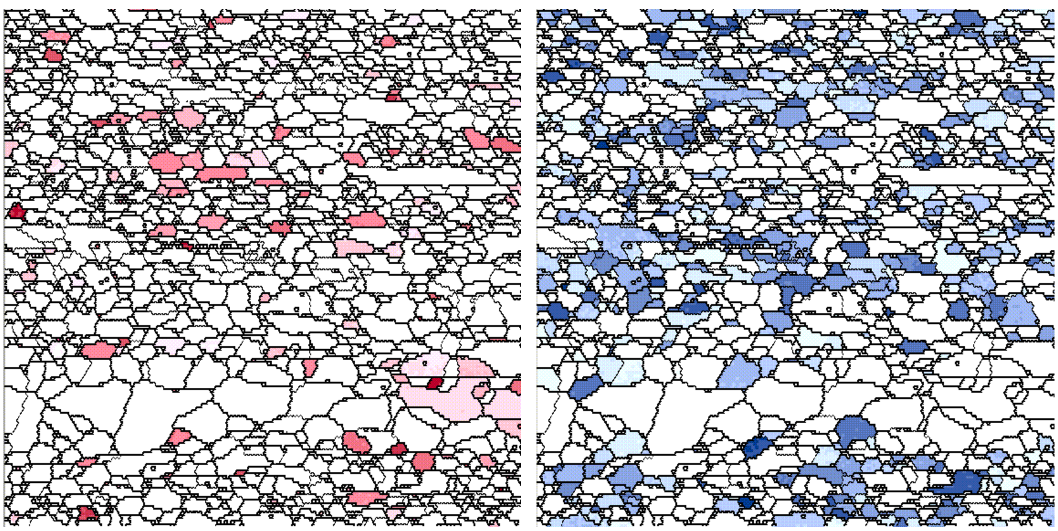


Figure 5. (a) <111> crystal direction map and (b) <100> crystal direction map showing a severe texture gradient from near the surface (at the top) to near the centerline (toward the bottom). This and other studies [6,7] using OIM to characterize unalloyed tantalum clearly indicate that surface x-ray measurements to characterize texture can be misleading due to the low penetration of the x-rays (typically less than 20μm).

Both optical metallography (Fig. 1), and the OIM scans reveal various extents of grain-size banding. Several OIM scans were performed in an attempt to correlate grain-size banding with low index crystal direction normals. These scans showed no evidence of strong texture associated with grain-size banding, i.e., the texture within a band of grain size appeared to be essentially random.

3.2 Tantalum-Tungsten Alloys

The microtexture analyses performed on the tantalum-tungsten alloys followed the same procedures used to examine the unalloyed tantalum. Four OIM scans were performed on the Ta-2.5%W alloy: two starting at the surface and progressing toward the center and two others spanning the centerline. This alloy was found to have a substantially different texture character than the unalloyed tantalum. A <111> crystal direction map taken near the centerline, shown in Fig. 6(a), reveals a significantly lower fraction of <111> normals relative to unalloyed tantalum. Figure 6(b) shows the increase in <100> crystal direction normals relative to unalloyed tantalum. The Ta-2.5%W alloy also exhibited more prominent grain-size banding, but like unalloyed tantalum, the grain-size banding did not show any preferred texture. (Like unalloyed tantalum, analysis of the crystal directions within various large grain size bands did not indicate a correlation with any low index directions.) The pole figures calculated from the scans indicated the primary texture of the Ta-2.5%W alloy is a <100> cube texture with no indication of <111> fiber.



(a) <111> 500 μm (b) <100>

Figure 6. Ta-2.5%W (a) <111> crystal direction map and (b) <100> crystal direction map. The direction fractions have changed prominence; <111> = 0.092 and <100> = 0.326. The grain-size band does not exhibit a texture correlation, i.e. the grain orientations are random.

Two OIM scans were performed on the Ta-5%W alloy: one starting at the surface and progressing toward the center and another spanning the centerline. This data indicated that the Ta-5%W alloy has approximately the same microtexture characteristics as the Ta-2.5%W alloy, i.e., predominately <100> crystal direction normals with some <111> normals. The relatively limited OIM dataset on this material suggests there is somewhat less grain-size banding and texture banding than the Ta-2.5%W alloy. However, this is inconsistent with the metallography shown in Fig. 1, which shows the Ta-5%W alloy to have more grain-size banding. Similar to unalloyed tantalum and the Ta-2.5%W alloy, the grain size bands did not have any identifiable texture associated with them. The pole figures calculated from the OIM data indicated a relatively strong <100> cube texture, again, similar to the Ta-2.5%W alloy.

The OIM data on the Ta-10%W alloy indicated the microtexture was essentially the same as the Ta-2.5%W and Ta-5%W alloys based on two scans: one starting at the surface and progressing toward the center and another spanning the centerline. The maps indicated predominately <100> crystal direction normals with some <111> normals with no significant texture banding and only moderate grain-size banding. The pole figures generated using the OIM data reveal less of a cube and more fiber texture than the 2.5 and 5%W containing alloys, consistent with the x-ray work of Michaluk [8].

4.0 Discussion

Figure 7 shows the <111> and <100> direction fractions for areas near the surface and at the center of the plates as a function of tungsten concentration. This plot reveals two important results: (1) The pure Ta is dominated by the large fraction of <111> normals while the tungsten containing alloys are dominated by the <100> normals. (2) The texture of the tungsten containing alloys is relatively uniform while the unalloyed tantalum has a significant variation; the <111> direction fraction is approximately a factor of two higher at the center of the plate relative to the surface.

The reasons for the variations in texture, given the similar processing of the unalloyed tantalum and the tungsten containing alloys, are not understood. The absence of texture gradients, and the relatively uniform <100> texture in the alloys, appear to be independent of tungsten concentration. This suggests a fundamental change in the recrystallization behavior due to the presence of tungsten concentration in the range of 2.5 to 10 wt.%. Also, the <100> cube texture of the W alloys is common to fcc rolled and annealed plate.

Consistent with the conclusions of Wright et al.[6,7], the results of this study suggest that the texture gradients in the unalloyed tantalum, as illustrated in Figs. 5 and 7, are solely responsible for the non-uniform mechanical response of unalloyed tantalum, e.g., hourglassing of compression samples. Conversely, the uniform behavior of the tantalum-tungsten alloys is a consequence of the absence of texture gradients. The presence of significant banding of grain size does not correlate with either texture gradients or the hourglassing of compression samples. For example, some of the tantalum-tungsten alloys exhibited significantly more banding of grain size compared with unalloyed tantalum, however, compression samples did not exhibit hourglassing. Also, as indicated in Sections 3.1 and 3.2 the texture within a band appeared to be essentially random. Based on these observations, it is clear that grain-size banding, by itself, does not cause inhomogeneous mechanical behavior.

The results of this study also suggest that the non-uniform failure behavior of unalloyed tantalum under quasistatic and dynamic loading, e.g., multiple necks in tensile specimens, is due to the variation in texture in the through thickness direction, and perhaps more importantly, variations in microtexture that were observed in the various OIM scans performed at various locations along the plate.

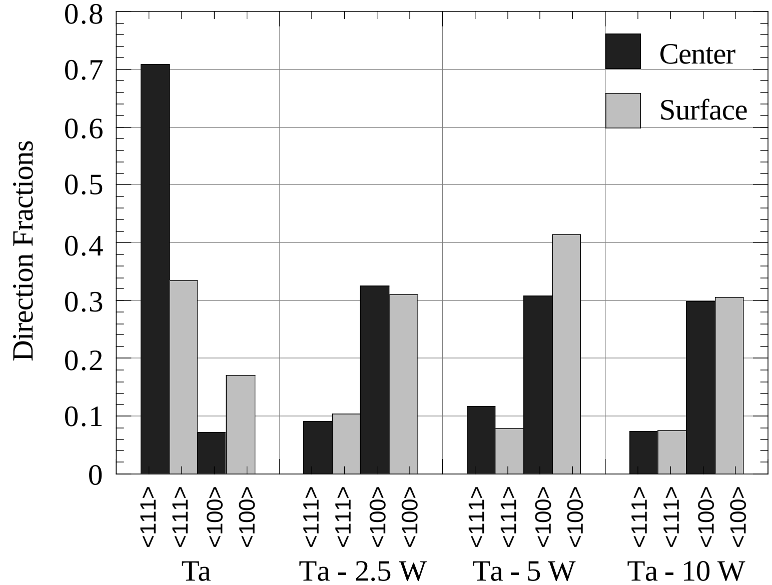


Figure 7. Crystal Direction Fractions as a function of tungsten content.

Acknowledgments

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